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THESIS

THE STRUCTURE AND BEHAVIOR OF VACUUM PLASMA SPRAYED OVERLAY COATINGS ON NICKEL BASED SUPERALLOYS

by

Patrick R. Norton

June 1983

Thesis Advisor: D.E. Peacock

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The Structure and Behavior of Vacuum Plasma Sprayed Overlay Coatings on Nickel Based Superalloys

by

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Lieutenant, United States Navy
B.S., Purdue University, 1977

Submitted in partial fulfillment of the
requirements for the degree of

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The feasibility of using the plasma spray technique for the application of metallic overlay coatings to marine gas turbine components was evaluated. Nickel based superalloy pins were sprayed with various MCrAl coatings using the plasma spray technique, given several types of surface treatments, and then oxidized at 1000 Degrees Centigrade for 100 hours. The effects of the various post-coating treatments on the coating structure and subsequent oxide behavior were investigated.

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I. INTRODUCTION AND BACKGROUND

A. MARINE GAS TURBINES

The gas turbine engine was initially developed to provide compact, high power to weight ratio engines for use in aircraft. As engine development progressed, the efficiency of the gas turbine began to increase substantially. The ability of the engine to reach full operating power very quickly opened new horizons for expanded utilization. Stationary power plants where high reliability, rapid startup, and high power levels are required began to utilize the gas turbine. The next logical step in the progression, was to introduce the gas turbine to marine applications.

In many instances, the marine gas turbine was not of a new design, but a modified or marinized version of an existing aircraft engine. The LM 2500 marine gas turbine, extensively used by the United States Navy, is a marinized version of the CF6 aircraft engine. This engine offers the features of light weight, ease of removal, and a modular parts exchange system. The first LM 2500 engines obtained by the Navy were installed on the container ship GTS Callahan. This was to become the U. S. Navy's gas turbine test platform.

Until this time, very little data was available on the performance of a gas turbine engine operating in a marine environment. The original aircraft version of the engine was operated predominantly at relatively high power levels and temperatures at high altitude. During the initial testing onboard GTS Callahan, a high power operating profile was utilized. During this period, performance of the LM 2500 was very good. Later, the operations of the ship were

modified to simulate the operating profile of a typical destrcyer. This prcfile included low power level operation together with a large number of transients, in contrast to the steady power level operation of a merchant ship, [Ref. 1].

B. SUPERALLOYS

The limiting factor in the successful operation of the gas turbine continues to be the ability of materials to operate in the high temperature, high stress environment of the gas turbine. The nickel based alloys IN 738 and Rene 80, originally develcped for aircraft applications, exhibit a high resistance tc creep and fatigue both in industrial power generation and marine environments, [Ref. 2]. However, the performance of currently available coatings for these alloys, again developed for aircraft engines, [Ref. 3], is proving to be inadequate in the marine environment, [Ref. 4].

C. CORROSION

Test data received from operation on the GTS Callahan indicated a new type cf corrosion was rapidly attacking the high pressure turbine blades. This attack was found to occur at the low temperatures associated with the low power level operating profile of a destroyer, [Ref. 5], and severly diminished the useable lifetime of the high pressure turbine when compared with the lifetime of similiar components in stationary power plant applications of the LM 2500 engine. The Navy, as well as civilian industry, initiated extensive research programs to identify the source of the degradation and to seek ways of improving blade life and reduce operating and maintenance costs. These efforts led to the development cf improved air filtration systems and

the use of MCrAlY overlay coatings, [Ref. 6], [Ref. 7], and [Ref. 8]. One such coating, BC-21, a CoCrAlY, is currently in use but improvements are still being sought.

D. COATING DEPOSITION TECHNIQUES

1. Physical Vapor Deposition

Presently, MCrAl overaly coatings are applied by Electron Beam Physical Vapor Deposition. This coating technique uses an electron beam to vaporize the coating material and deposit it on the airfoils. The coating builds up in long columnar grains, normal to the surface. The columnar grain boundaries, see Figures B.1 and B.2, are potential diffusion paths [Ref. 9] for the alloying elements; such diffusion can possibly degrade the coating performance. Research into the problems associated with coating-substrate inter-diffusion is currently underway at the Naval Postgraduate School. This technique also provides excellent bonding of the coating to the substrate, low porosity, (as shown in Figures B.3 and B.4) and cost effectiveness.

2. Plasma Spray Technique

Alternate methods of applying metallic overlay coatings are continually being sought as a means of improving coating performance and reducing the costs of the coating process. The plasma spray (PS) technique appears to be one possible means of achieving these aims. It has the ability to apply a wider range of coating compositions, has lower capital and lower application costs and has the ability to provide much higher depostion rates, when compared with physical vapor deposition. In addition, unlike PVD, the plasma spray process also has potential as a method for selectively re-coating overhauled turbine blades, an ability which could substantially reduce lifecycle costs.

There are several variations of the plasma technique. All essentially spray powdered metal into an inert gas plasma, which in turn is sprayed onto the component. The components can either be sprayed in air using an inert gas shielded torch or the parts can be placed in an inert gas chamber at atmospheric or reduced pressure. The advantage of a reduced pressure chamber is the ability to achieve very high plasma velocities. These can range from subsonic to as high as Mach 3, [Ref. 10].

The two vendors that supplied samples for testing in this study used vacuum plasma spray equipment developed by different manufacturers. While the actual spraying process is basically the same for different systems, there are many small variations in spray parameters, often proprietory, which can produce substantial differences in the structure of the applied coating. Other parameters, such as powder size, age of the powder, precoating cleaning procedures, and deposition temperatures, also effect the structure of the coating. Presently, the vendors are heat treating the sprayed coating to provide better adherence of the coating to the substrate.

The PS coating technique builds up material on the substrate in overlapping layers as shown in Figure B.5. The resulting grain structure, shown in Figure B.6, contains fewer short-circuiting diffusion paths than are apparent in the columnar grain structure of a PVD coating.

Research conducted has shown that it is difficult to maintain consistent quality throughout the spray process. In addition, problems have been encountered concerning porosity, lack of coating adhesion, and surface roughness. [Ref. 11].

E. OXIDE FORMATION AND BEHAVIOR

Materials used to manufacture gas turbine blades are protected from high temperature oxidation through the use of coating systems which form a protective oxide layer. In service, oxidation of the coating continues to occur until the surface is effectively sealed from the environment by the oxide itself. If the oxide has a tendency to spall during service, the oxidation process continues to be repeated until the coating is depleted, and all protection to the turbine blade is lost, [Ref. 12]. Additionally, an effective oxide layer is one which forms a uniform oxide thickness so as not to alter the overall aerodynamic profile of the blade. The oxide must be thin so as not to reduce the airflow through the blading.

The oxide which subsequently forms from plasma sprayed coatings does not meet the above requirements. The as sprayed surface is very coarse as indicated in Table III. Various post-coating treatments will produce an acceptable surface finish. All of these methods modify the surface and/or the structure of the coating and may alter the structure of the oxide formed at high temperture.

F. OBJECTIVES

The purposes of this study are to evaluate the present state of the art in plasma spray technology and to investigate the effects of various post-coating treatments and combinations of treatments on surface finish and on subsequent oxide formation, structure and adherence.

II. EXPERIMENTAL PROCEDURE

A. SAMPLE AQUISITION AND PREPARATION

Samples of four different coating-substrate systems in the form of 1/4 and 1/2 inch cylindrical pins, were obtained from commercial sources. These are summarized in Table I in terms of the substrate alloy designation and coating composition. All four coatings were applied by the reduced pressure inert gas process. The post-coating surface treatments employed are summarized in Table II. To develop a surface oxide layer, samples, including those in an as-sprayed condition, were heated in static air. Investigative techniques employed included surface profilometry, optical microscopy, and scanning electron microscopy.

B. SPECIFIC TEST PROCEDURES

1. Surface Finish Analysis

The surface finish of the coating was determined by a profilometer machine. The sensitivity setting for the as-received and peened sample measurements was 50 micro-inch per division. The sensitivity was reduced to 10 micro-inch per division for polished samples.

A short section of each pin was deliberately left uncoated and the thickness of the coating was determined by measuring the diameters of the uncoated and coated sections with a micrometer.

2. Oxidation

Oxidation was carried out by exposure to air at 1000 deg. Centrigrade for 100 hours in a laboratory furnace. Specimens were furnace cooled to below 100 deg. C. before removal. Resistance to cyclic oxidation was not evaluated.

3. Polishing

Polishing was accomplished by spinning the sample and abrading the surface with silicone carbide abrasive paper. Two types of polishing were performed. The first consisted of polishing at 500 rpm with 320 grit paper for approximately 2 minutes. The main objective here was to remove any loose particles on the surface of the coating, which was a common feature in some cases.

The second type consisted of polishing with progressively finer and finer paper, starting with 240 and following with 320, 400, and finally 600 grit.

4. Shot Peening

Peening was performed at a commercial installation, utilizing glass bead shot. Peening was not utilized as a method of densifying the surface but as a method of modifying the structure of subsequently formed oxides. A heavy (12-16N) shot intensity was achieved using an automatic shot peening machine with the samples mounted in holders. A light (6-8N) shot intensity was achieved using a hand operated gun.

5. Metallographic Techniques

a. Mounting and Polishing

Standard mounting and polishing techniques were used to prepare samples for metallographic examination. Care was taken to use a mounting material stable in a bromine solution.

b. Etching Procedures

(1). Krcll's Reagent. Kroll's Reagent is a solution cf hydroflcuric and nitric acid in water, which brings out the grain structure of the coating, [Ref. 13]. Etching times ranged from 1 to 2 minutes.

(2). Deep Etch Technique. Deep etching [Ref. 14] is a comparitively new techique for determining the structural features of coating-oxide systems, particularly the underside cf the oxide at the oxide/coating interface. A cross section of the oxide-coating-substrate system is mounted and polished as for metallographic observation.

The deep etch reagent consists of a solution cf 10 ml of reagent grade liquid bromine combined with 90 ml of absolute, low acetone, methanol.

The sample is inverted in the bromine etchant and slowly swirled to allow the metal particles to fall cff as the metal is etched. Etching times range from approximately 1 to 4 minutes. After etching, the samples are rinsed by slowly swirling in a series of beakers containing absolute methanol. Five rinses are recommended. No discclcration of the methanol in the last two beakers indicates sufficient rinsing. Care must be taken to prevent the delicate, exposed oxide from being damaged.

Samples thus prepared can be observed in the Scanning Electron Microscope. The SEM has sufficient depth of field to allow all of the exposed oxide to be viewed at one time. If static charge build-up on either the oxide or the mounting material becomes a problem, the application cf a vapor-deposited coating of carbon or gold is recommended.

Very careful observations must be made to identify micro-pegs, the characteristic feature of a well-bonded oxide. These are small protuberances on the surface

of macrc-pegs and are a feature of coatings which contain an "active element" such as hafnium or lanthanum. Macro-pegs result frcm the growing oxide following the normally rough surface of the coating and are apparently insufficient by themselves to prcduce a good bond. The surface of the oxide will replicate the coating contours and a false assumption of oxide adherence can be made. Figures B.7 and B.8 illustrate the appearance of macro and micro pegs.

III. RESULTS AND DISCUSSION

A. RESULTS

1. Coating Structure

a. Vendor "A"

The structures of both the NiCrAl and CoCrAl type coatings supplied by this vendor were similar. Figure B.6 is a typical example of the excessive porosity encountered in all parts of every coating. The amount of porosity appears greatest at the coating/substrate interface. Figure B.9 illustrates a complete lack of bonding of the coating which was another common feature. The grain structure in general appeared coarse and nonuniform.

Figure B.10 is a typical surface profile of an as-sprayed coating. The results of the surface finish measurements are shown in Table III. There is a high degree of variation in the surface finish. There exist large surface voids next to areas of what appear to be loosely adhered particles of coating material, as shown in Figures B.11 and B.12.

b. Vendor "B"

Similar problems were experienced with this vendors products, but to a much lessor degree. Generally, the coating structure was fine grained and uniform. Bonding of the coating to the substrate appeared to be generally good. Only a few localized regions of porosity were found. Figure B.13 shows a typical cross section which illustrates the much more uniform surface structure of this vendors coatings. Figure B.14 is a typical surface profile; results are tabulated in Table III.

However, there are isolated cases of severe defects in Vendor "B"'s coatings. Figure B.15 is an example of an islclated case cf cracking. Figure B.16 shows a large void exists in the ccating which reduces the local coating thickness to essentually nill. It is difficult to determine if the cracking occurred during the coating process or during pcstcoating processing and analysis. Samples were held in nylcn chucks to minimize the possibility of introducing defects during cutting. Figure B.17 is a typical sample illustrating a fine grain structure and low porosity.

2. Effects of Pclishing

The second type of polishing treatment left an essentially uniform 600 grit finish. The amount of material that had to be removed to produce a uniform 600 grit surface finish ranged from 15% for the fine grained coatings to as much as 50% for the course grained coatings. Samples examined in the SEM showed that polishing did reach all portions of the surface althcugh some low points did remain. An excessive arcunt of ccating would have to have been removed to ccmpletely eliminate these low points. Table III lists the results of surface profile measurements taken before and after pcst-ccating pclishing treatments.

Viewed in SEM, the surface of samples oxidized after polishing appeared tc be very similar to those of samples oxidized in an as-sprayed condition. A coarse grained coating produced a ccarse oxide structure. In addition, the polishing did not appear to effect the bonding of the cxide. Figure B.18 is an example of a polished and cxidized specimen. A coarse cxide has been produced and has caused subsurface oxidation of the coating. This subsurface oxidation reduces the effective thickness of a coating and thus the operating lifetime of a component. The oxide produced cn coatings containing hafnium appeared to be bonded. The

oxide on those without hafnium was poorly bonded and was lost during the heat treatment or subsequent handling.

3. Effects of Shot Peening

Figure B.19 is an example of a shot peened surface of an as-sprayed sample. The peening has smoothed the high points of the surface, but left the low points essentially untouched. The oxidized sample, Figure B.20 shows loose oxide at the low points of the sample, and fine grained oxide at the high points. The oxide produced at the low points is similar to that found on as-sprayed samples.

Figure B.21 shows an oxidized sample with severe cracking in the coating and separation of the coating from the substrate.

The purpose of peening is to modify the coating structure in such a way that subsequently formed oxide is effectively bonded. As shown in Figure B.22, the coating grain size was slightly reduced at the surface from shot peening. Based on examination of oxidized samples, the shot peening application did not appear to alter the bonding of the oxide, it did however, appear to produce a thinner and finer oxide structure.

4. Combination Treatments

a. Shot Peening followed by Polishing

Shot peening the as-sprayed surface effectively worked only the high points of the surface. Subsequent mechanical polishing to produce a smooth surface simply removed these worked regions. The net result was as if the sample was only mechanically polished and no effect of shot peening was evident in the oxide structure.

b. Polishing Followed by Shot Peening

The oxide produced by this combination of treatments was very fine grained and uniform over the entire surface. The polishing gave a smooth surface which could then be evenly shot peened. The uniformity of the structure is dependent on the requirement that all portions of the coating surface receive the same treatment. Figures B.23 and B.24 are examples of fine grained coatings which were polished, peened and then subsequently oxidized. The shot peening appears to produce a finer oxide structure than could be achieved with a polishing treatment alone. The oxide produced in the latter case appears to be directly related to the grain size of the as-sprayed coating. Shot peening in both cases appears to make the structure of the resultant oxide finer. This indicates that shot peening does have a direct effect on the structure of the oxide produced. The above treatments had no apparent effect on the ability of the oxide to bond to the coating. The oxide was tightly bonded on the samples which had hafnium in the coating.

The coarse grained coatings were given a heavy shot peening which deeply scarred the surface, (Figure B.25). The oxide produced by this treatment was not uniform, and was similar to the oxide produced on the as-sprayed samples. An additional polishing step was done in an attempt to achieve a uniform surface. Subsequent oxidation produced a nonuniform oxide structure since the high points remaining after the peening operation, which were worked and would have resulted in a fine oxide structure, were removed by polishing.

5. Active Element Effect

The presence of micro-pegs was used as an indication as to whether the active element in the coating formulation was actually involved in the oxide formation. In most cases in which the active element was not present, the outer layer of the oxide was so fragile that is would spall off during cooling after heat treatment or during sample preparation. Dot mapping scans were done for cobalt, chromium and aluminum. The scans showed that the large flakes in Figure B.26 are rich in aluminum, (Figure B.27), and deficient in cobalt and chromium. Coatings which contained hafnium showed the presence of micro-pegs in the oxide at the oxide/metal interface when viewed in the SEM after deep etching, Figures B.28 and B.29. In the coatings to which lanthanun was added, no active element effect was evident, in that no micro-pegs could be seen, Figures B.30 and B.31.

B. DISCUSSION

This investigation has shown that the plasma spray process, when carefully controlled, is capable of producing fully dense and adherent overlay coatings. If the process is not carefully controlled there is a tendency for unacceptible porosity to occur, both within the coating and at the coating-substrate interface. Voids at the interface cause the coating to separate from the substrate when the system is subjected to thermal cycling. Voids located near the surface allow internal oxidation to occur which undercuts the surface and causes large sections of non-oxidized material to be removed. The as-sprayed surface of the coating appears to be inherently rough and the coating itself can be coarse. Post-coating treatments are required so that the oxide formed on subsequent exposure to high temperature is free from surface flaws and fine grained.

This type of oxide, together with micro-peg formation from the active element effect, ensures satisfactory oxide performance during service.

1. Post-Coating treatments

a. Polishing

Polishing needs to be carried out until a smooth surface is obtained. The amount of material to be removed is simply related to the roughness of the as-sprayed coating. The uniformity and structure of the oxide formed on polished samples always appeared to be similar to that of the oxide formed on the as-deposited structure. A surface smoothing operation of some type is required, subsequent to coating, and polishing has been found to be satisfactory for this purpose. A smooth surface is also required for a satisfactory peening operation.

2. Glass Bead Peening

Shot peening leads to a refinement of the structure of the coating at the surface. There is some evidence that this refinement results from recrystallization of the cold-worked layer on exposure to high temperature, [Ref. 15]. This refinement has a beneficial effect on the structure of the subsequently formed oxide. Satisfactory shot peening requires that the initial surface be relatively smooth. Shot peening is not capable of smoothing a very rough surface and any attempt to increase the rate of smoothing by increasing the peening intensity is likely to be counterproductive. Polishing followed by glass bead shot peening appears to produce the best oxide structures.

3. Active Element Effect

The formation of a smooth, uniform coating and oxide along with the ability of the oxide to become adherently bonded to the coating are necessary requirements for satisfactory oxide performance. The active element lanthanum was in the coating formulation used by Vendor "A" while some of the samples received from Vendor "B" contained the active element hafnium. The deep etch technique revealed the presence of micro-pegs on the underside of the oxide on the coatings containing hafnium, but there was no indication of micro-peg formation for the samples containing lanthanum. In fact, the oxide could be seen spalling off the samples when they were removed from the furnace after oxidation. It is likely that the lanthanum was prematurely oxidized during the coating process and was unable to participate in the formation of the oxide.

IV. CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

The vacuum plasma spray technique can be used to apply acceptible metallic overlay coatings to nickel based superalloys. Combining this process with post-coating treatments of polishing and shot peening can produce smooth, uniform oxide structures when exposed to high temperatures. Further, an adherently bonded oxide (through micro-peg formation) can be produced if the coating formulation contains an active element, hafnium in this study. Care must be taken to prevent premature oxidation of an active element through lack of control during the coating process.

B. RECOMMENDATIONS

1. Vendors

Additional work must be done to decrease the level of porosity and increase the bonding between the coating and substrate. The ability to maintain a consistent quality throughout successive coating runs must be demonstrated. In particular, care must be taken to use good quality powders.

2. Additional testing required

a. Post-Coating Treatments

The range of treatments utilized after coating could be expanded to include slurry polishing and tumbling in a ceramic medium. Currently, as-sprayed coatings are heat treated immediately following the coating process. If this is not done, it is found that the coating is easily removed during subsequent handling. A possible alternative

treatment might to perform shot peening or hot isostatic pressing. These may decrease porosity and increase coating-substrate bonding. [Ref. 16].

b. Active Element Effects

Additional coating formulations need to be acquired to determine if elements other than hafnium can lead to an active element effect in plasma sprayed coatings.

c. Oxidation and Corrosion Testing

The testing program needs to be expanded to include laboratory cyclic oxidation and hot corrosion evaluation.

APPENDIX A
TABLES

TABLE I
Coating Composition

		Vendor "A"		Vendor "B"	
Substrate		IN738LC	IN738LC	Rene 80	Rene 80
Chromiun	%	34.0	30.0	26.52	25.46
Aluminum	%	7.0	9.0	11.30	10.30
Tantalum	%	2.5	2.5	---	---
Manganese	%	1.7	1.7	---	---
Lanthanum	%	1.0	1.0	---	---
Hafnium	%	---	---	---	3.14
Oxygen	ppm	---	---	430	---
Nickel	%	Bal	---	---	---
Cobalt	%	---	Bal	Bal	Bal

TABLE II

Post-Coating Treatments

1. Mechanical Polishing
2. Glass Bead Shot Peening (light / heavy)
3. Polishing / Peening
4. Peening / Polishing
5 Polishing / Peening / Polishing

TABLE III

Typical Surface Profiles

	Vendor "A" (microinch)	Vendor "B" (microinch)
as sprayed	750-1600	400-800
shot peened	150-200	50-100
polished	<10	<10
polished/peened	50-100	40-60
pol/peen/pol	<10	<10

APPENDIX B
FIGURES

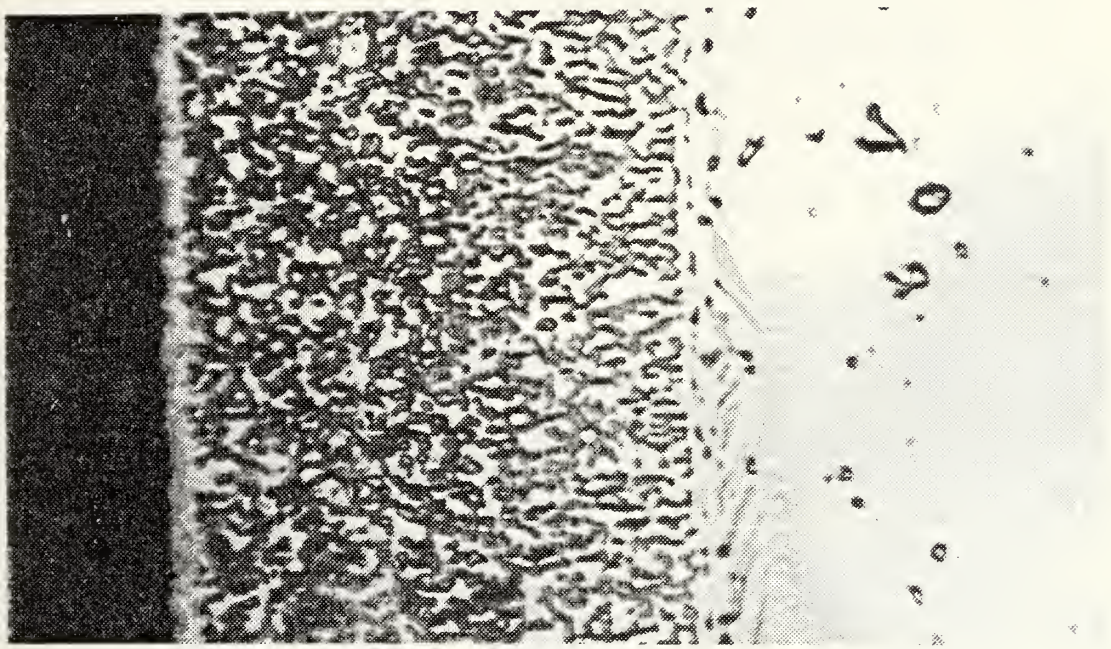

Figure B.1 PVD deposited coating showing excellent coating adhesion, uniform structure and lack of porosity. Etched, 680X

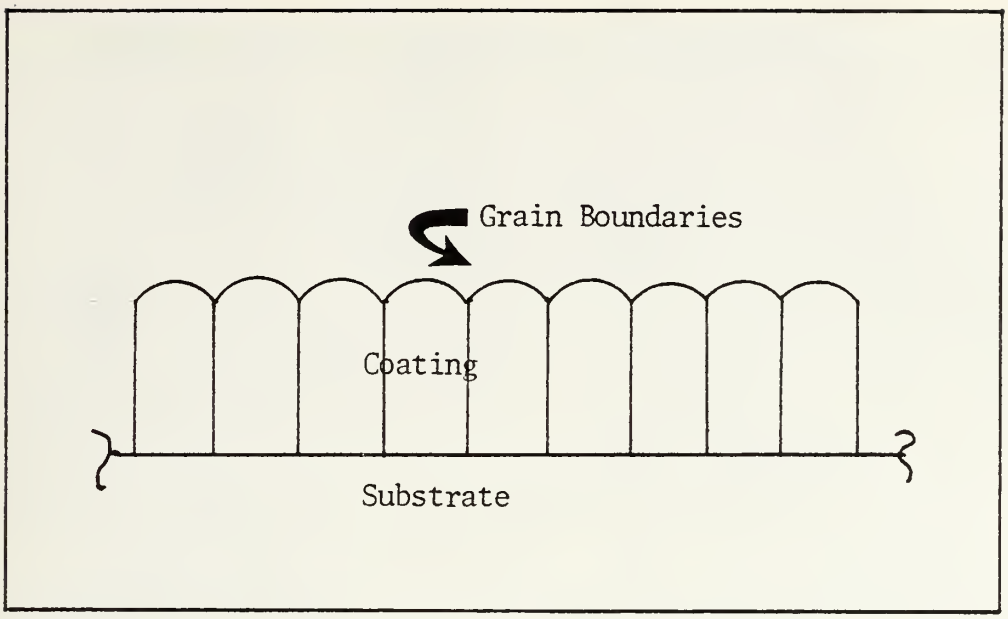

Figure B.2 Illustration of EB-PVD columnar grain structure

Figure B.3 Columnar grain structure of BC-21. 680X

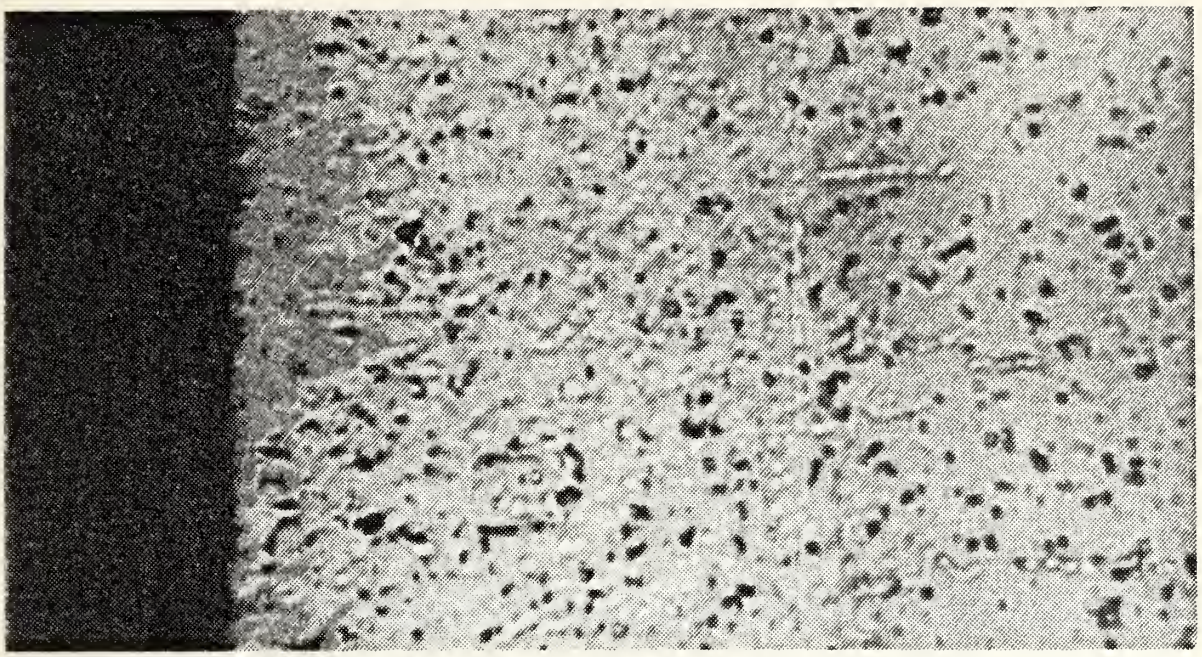

Figure B.4 BC-21 on Re-80 showing good bonding of coating. 680X

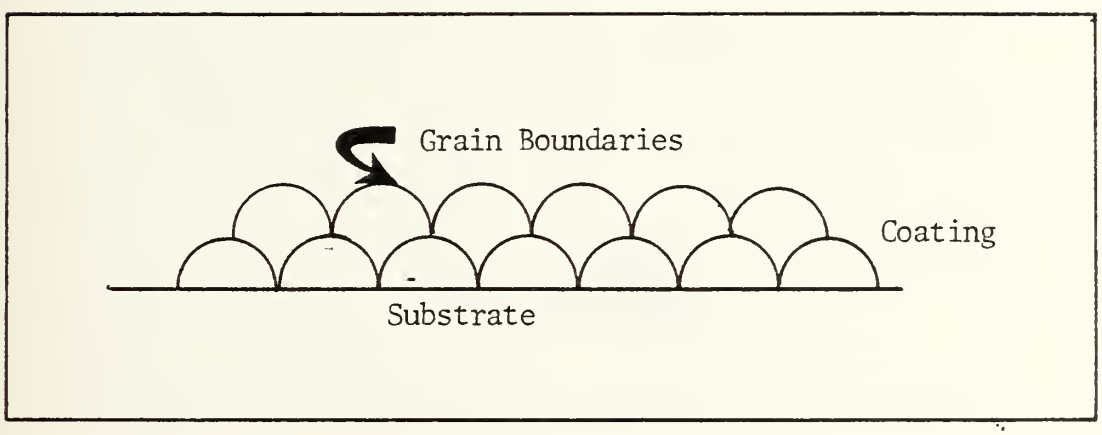

Figure B.5 Illustration showing the overlapping grain structure of PS deposited material.

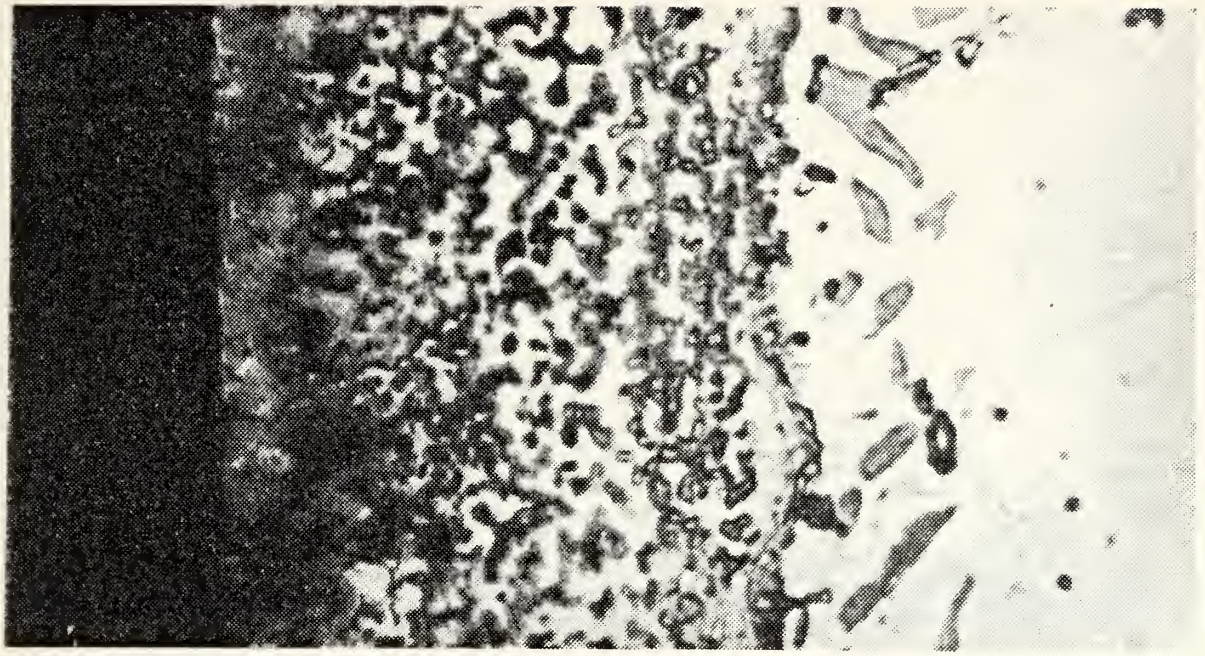

Figure B.6 Example of plasma sprayed coating exhibiting a high degree of porosity. Etched, 680X

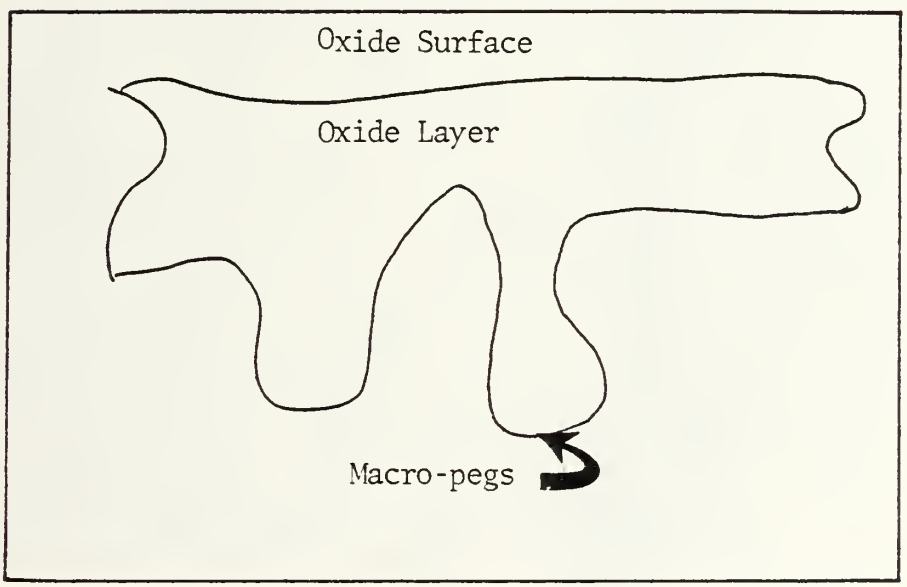

Figure B.7 Illustration of macro-pegs.

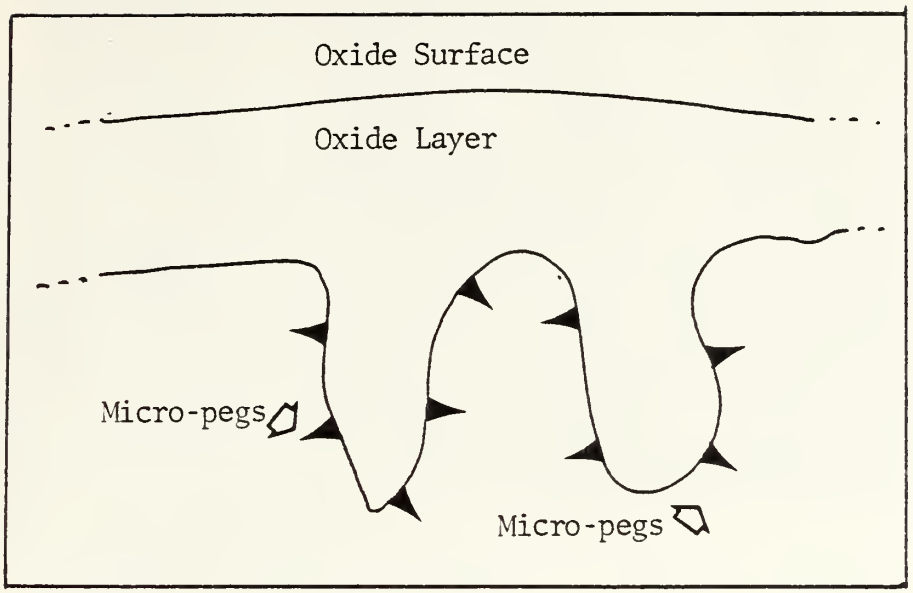

Figure B.8 Illustration of micro-pegs.

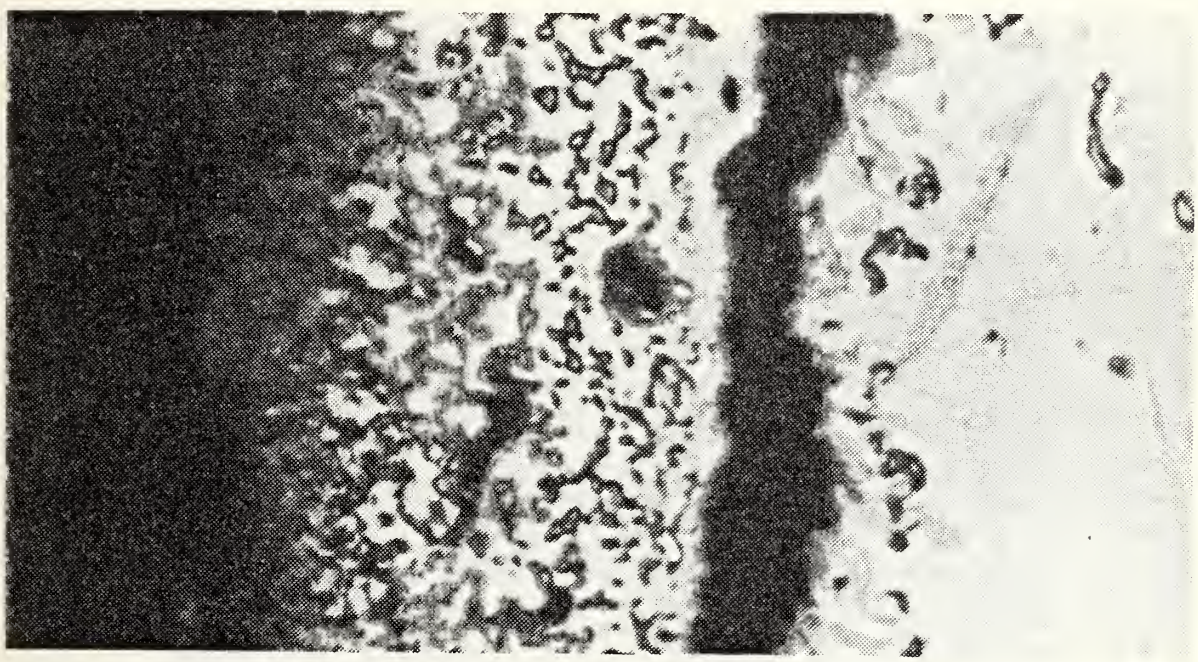

Figure B.9 Plasma coating applied by Vendor "A" showing complete separation of the coating from the substrate. Etched, 850X

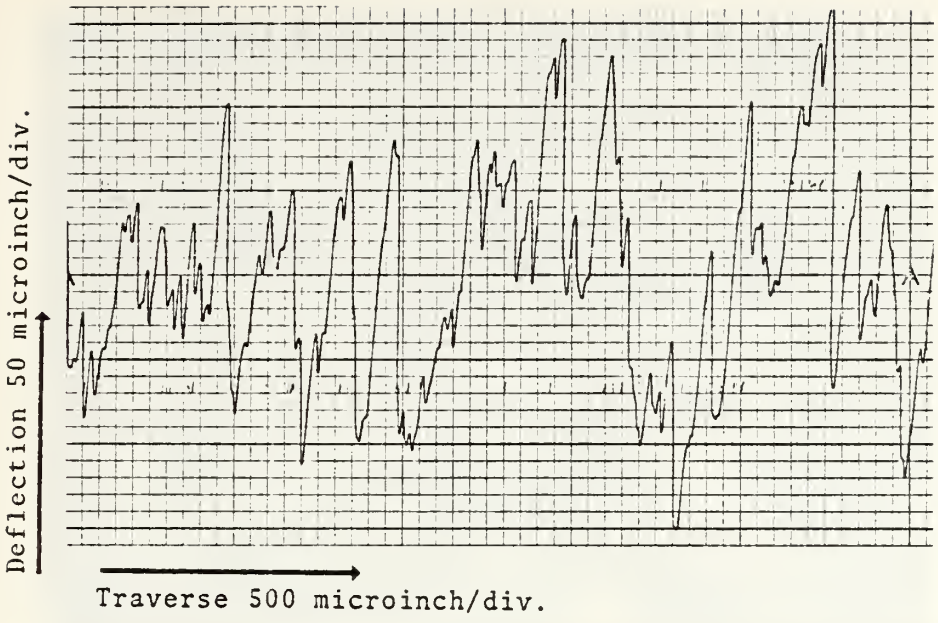

Figure B.10 Typical surface profile of plasma coating applied by Vendor "A".

Figure B.11 Example of an uneven surface and loose particles exhibited by a plasma sprayed CoCrAl coating. SEM, 1000X

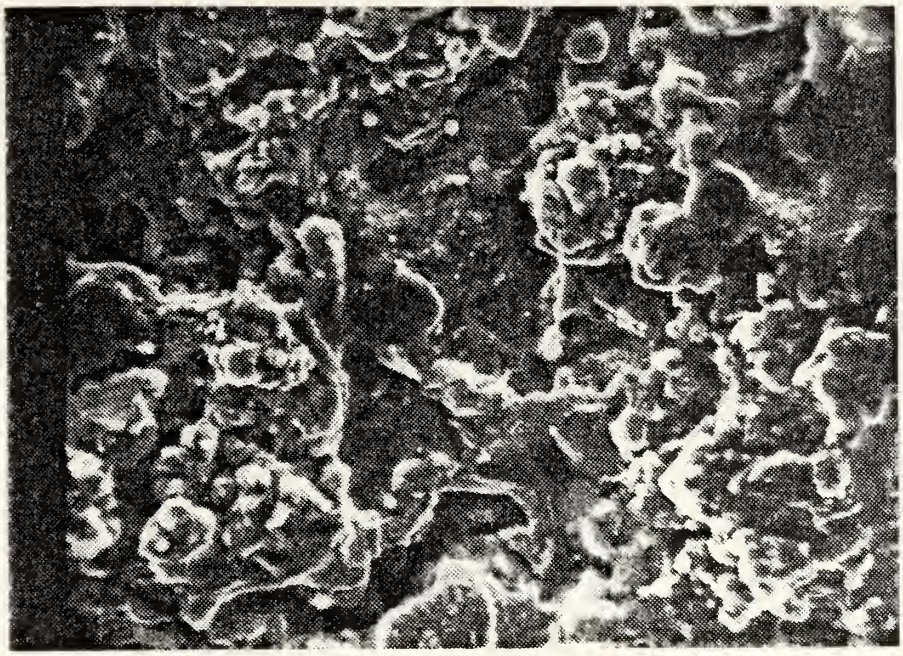

Figure B.12 Example of an uneven surface and loose particles exhibited by a plasma sprayed NiCrAl coating. SEM, 1000X

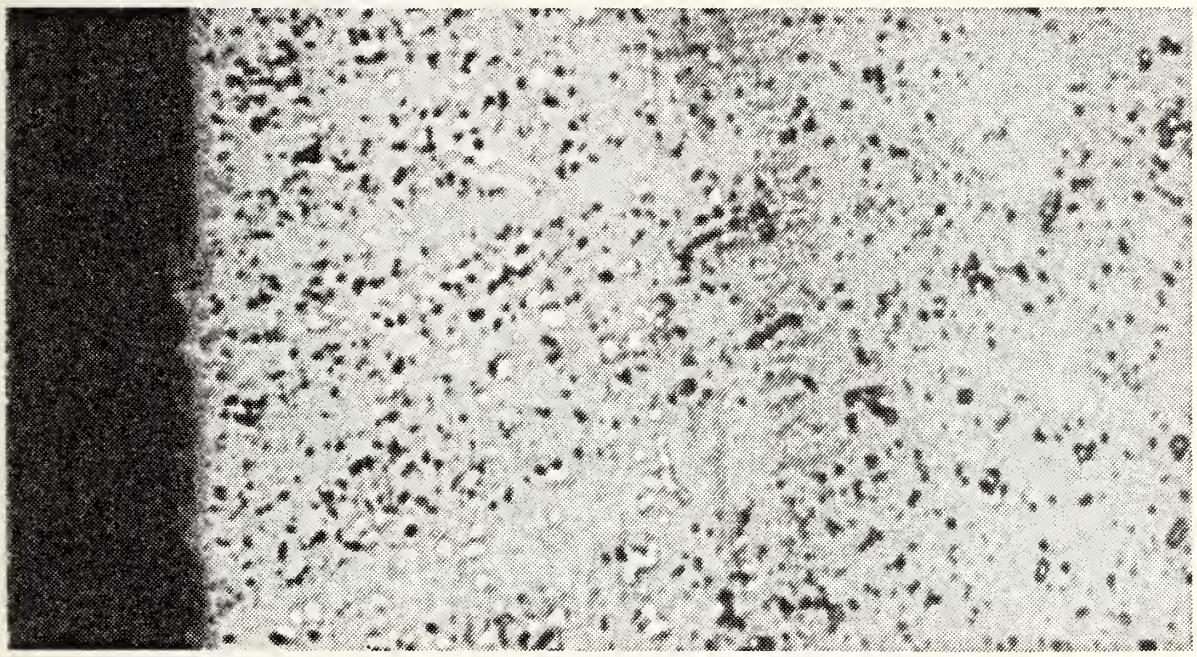

Figure B.13 Plasma sprayed coating by Vendor "B" with low porosity and good coating/substrate bonding. 680X

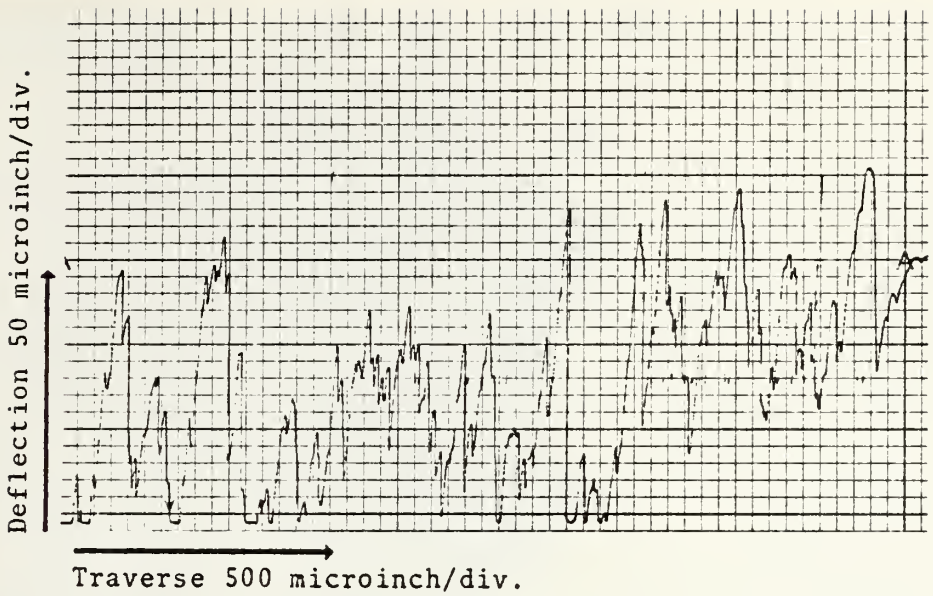

Figure B.14 Typical surface profile of PS coating applied by Vendor "B".

Figure B.15 Small area of localized cracking of a PS coating applied by Vendor "B". 680X

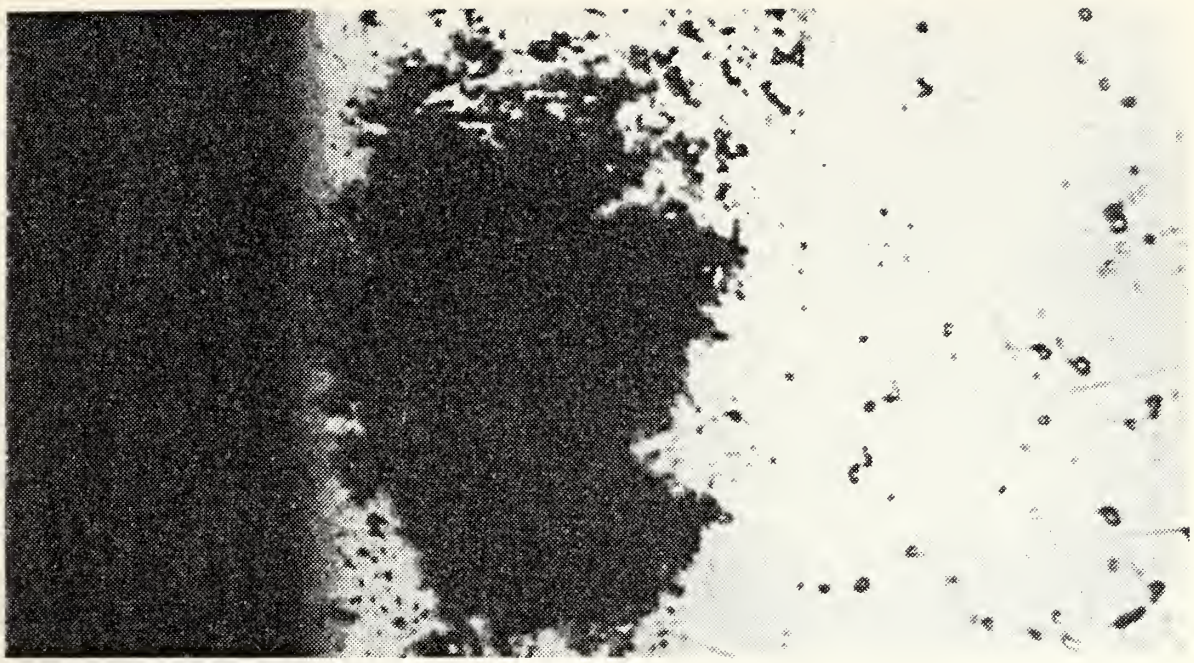

Figure B.16 PS coating applied by Vendor "B" exhibiting a a large subsurface void. The void extends through most of the coating thickness. 850X

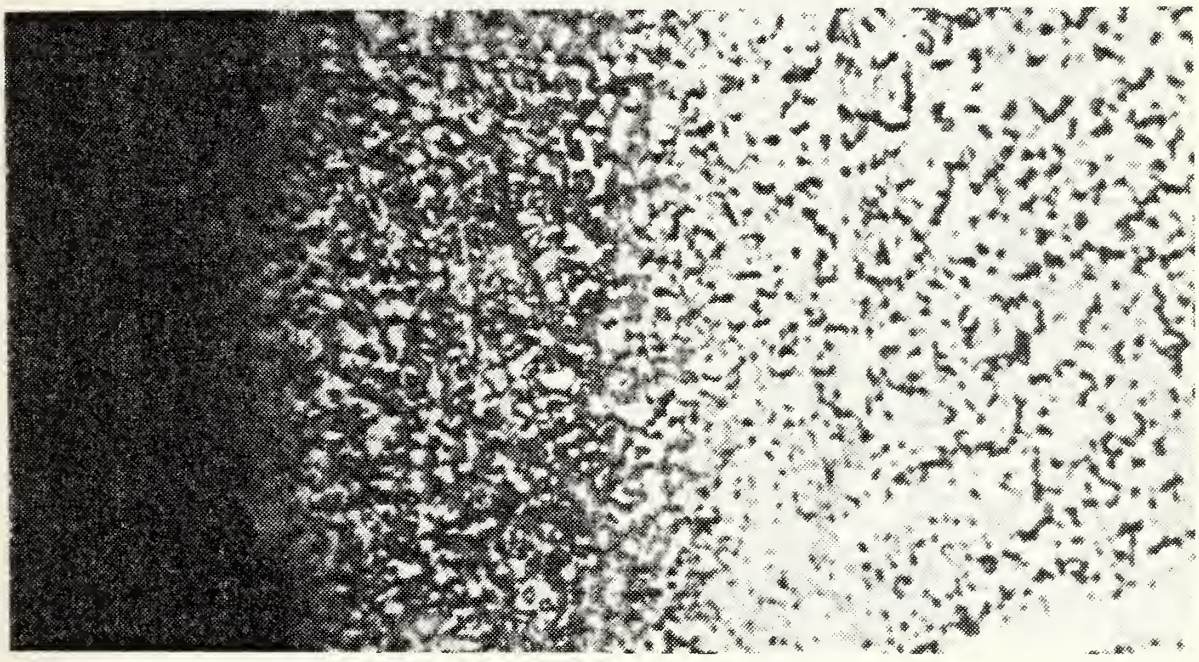

Figure B.17 Oxidized sample from Vendor "B" with good coating to substrate adhesion. 680X

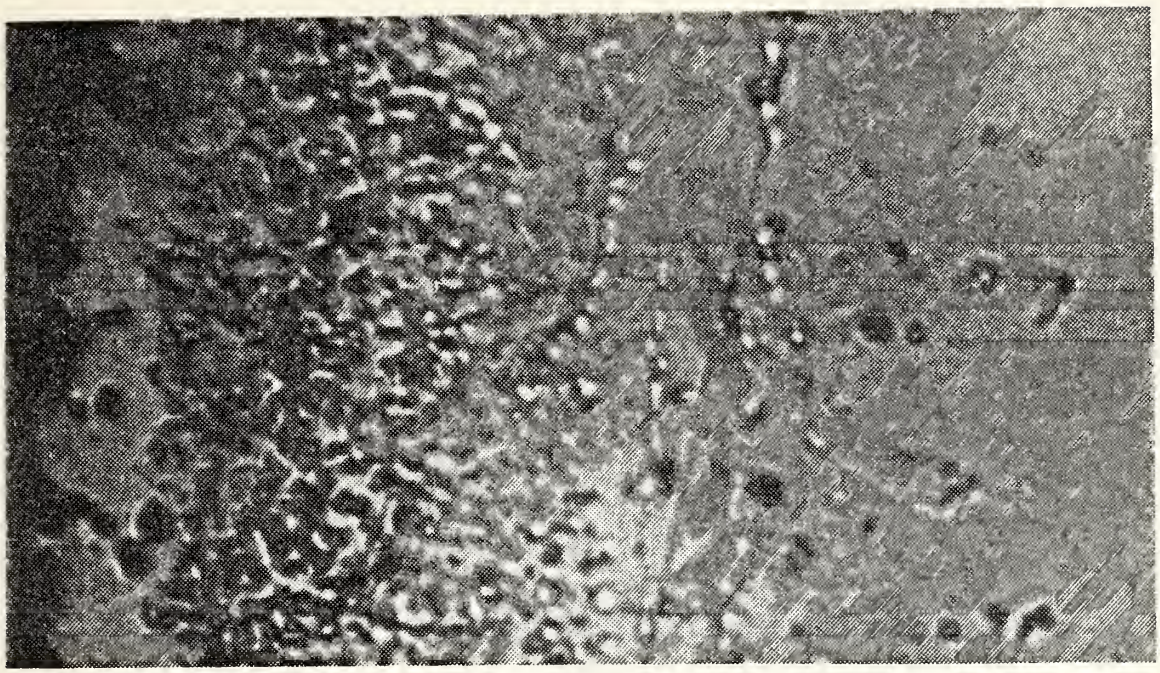

Figure B.18 Polished and oxidized sample showing oxide undercutting the coating surface. 850X

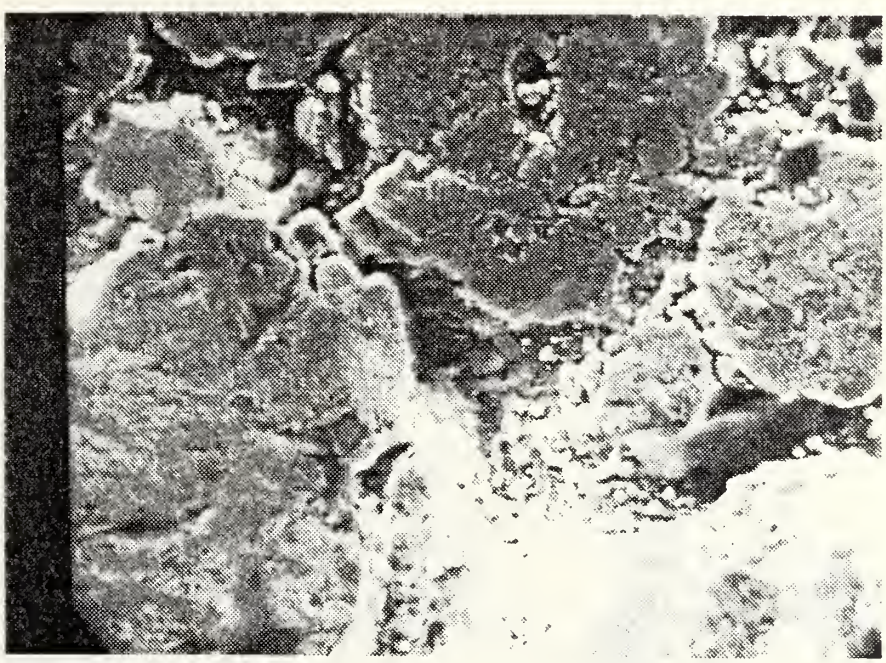

Figure B.19 Peened surface of coating illustrating the inability of peening to produce a uniform surface. SEM, 1100X

Figure B.20 Peened and oxidized surface of sample showing uneven oxide formation with large, unbonded particles of oxide in the depressions. SEM, 1200X

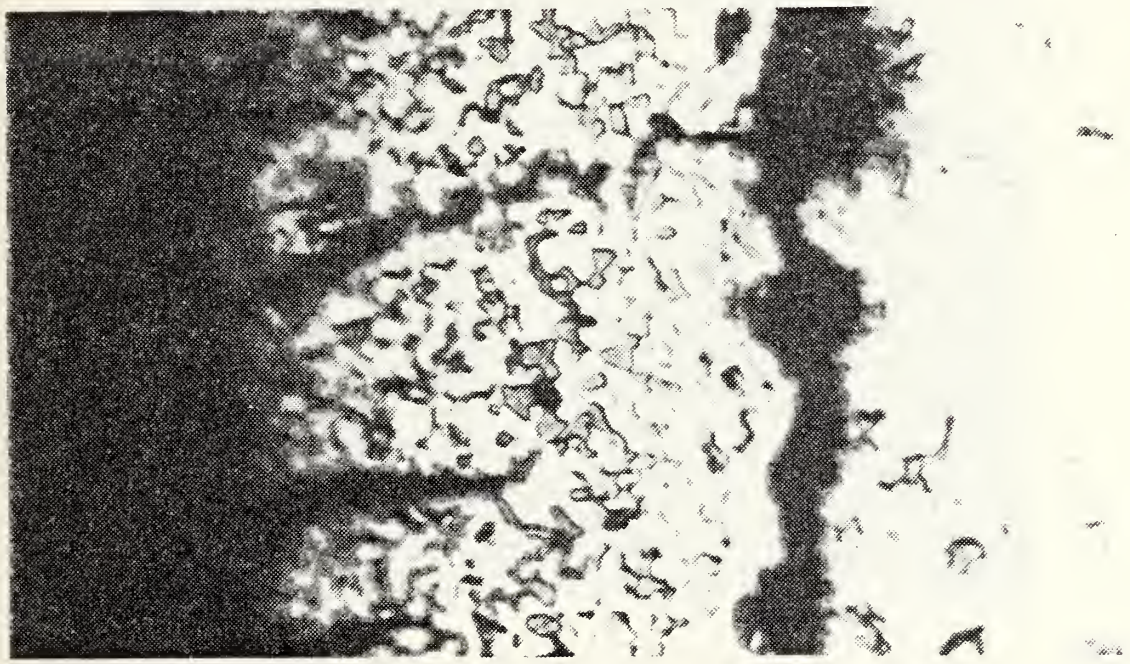

Figure B.21 Severe cracking and separation of a peened and oxidized coating. 850X

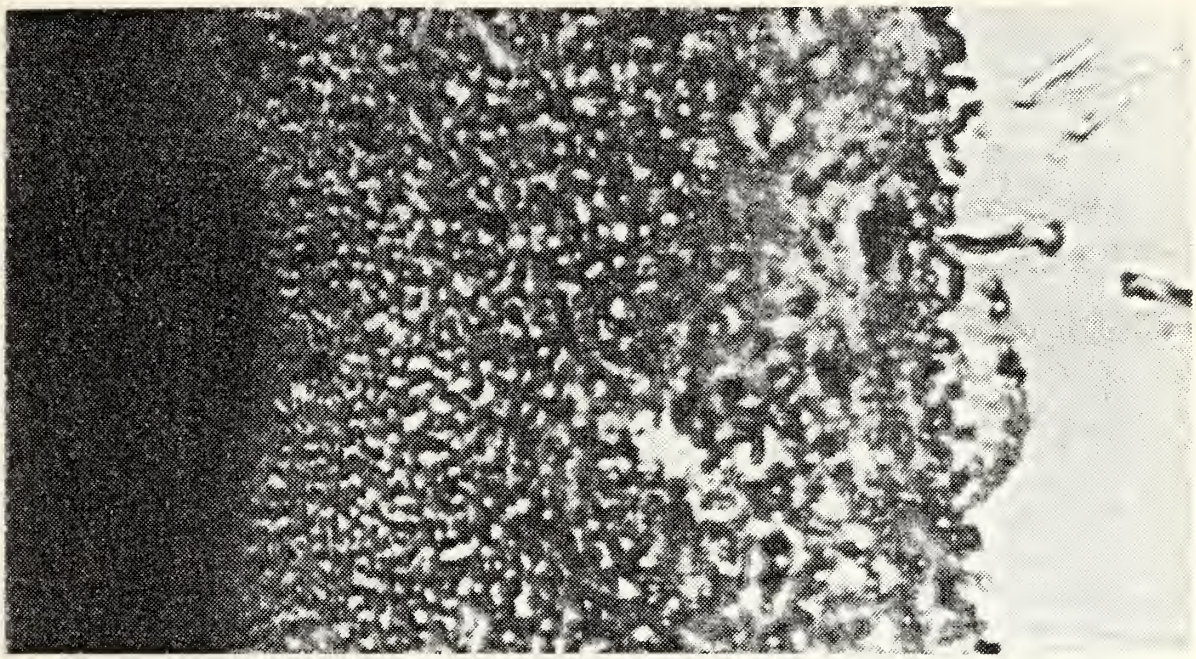

Figure B.22 Fine grained structure at the surface of a peened coating. 850X

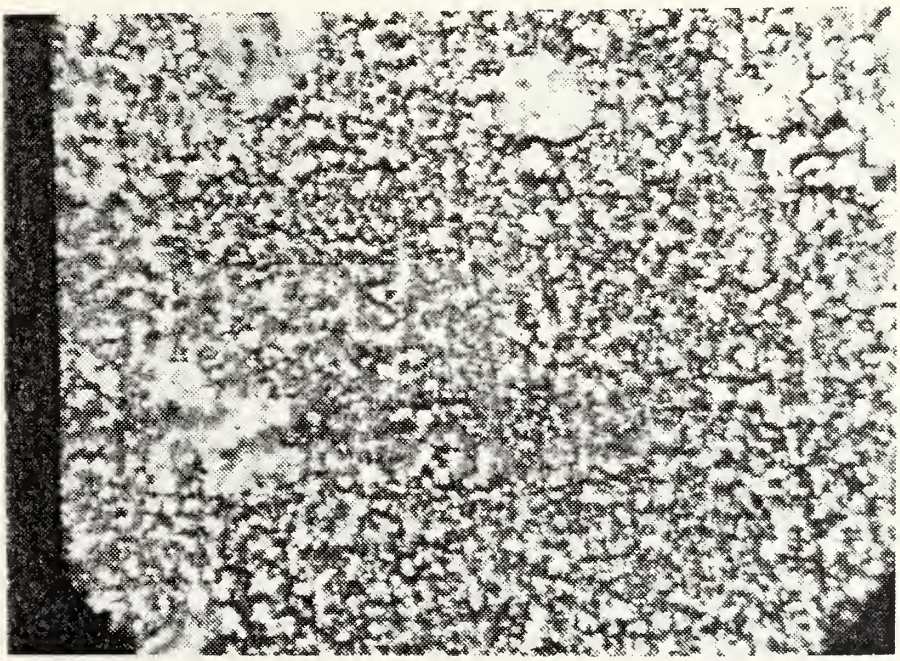

Figure B.23 A polished, peened and oxidized sample showing a smooth surface and a uniform oxide structure. SEM, 1200X

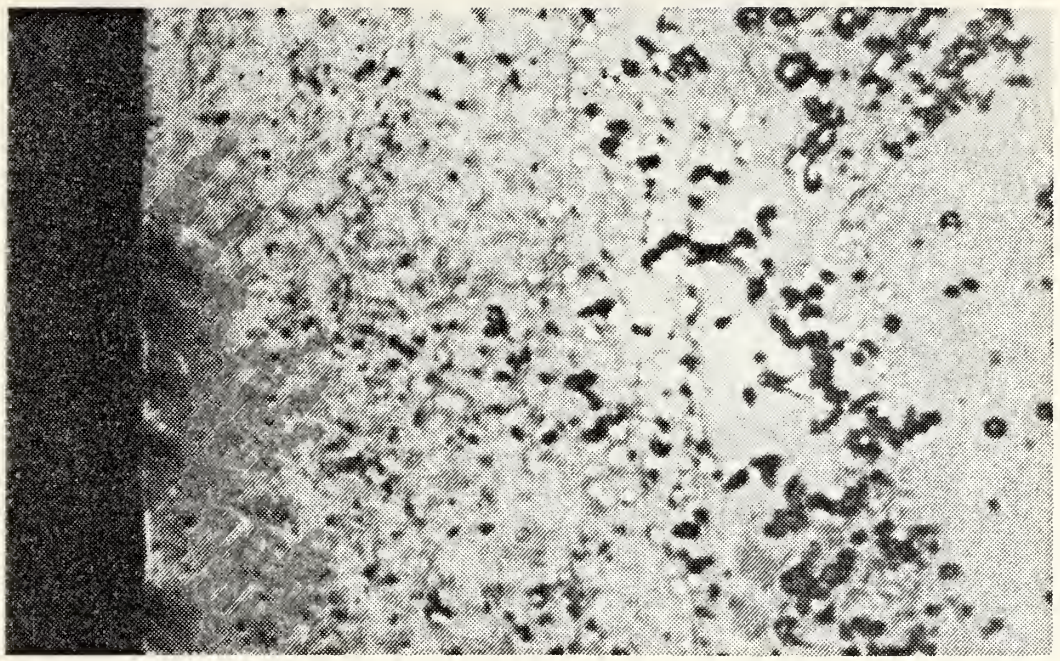

Figure B.24 A polished, peened and oxidized sample showing a smooth surface, a uniform oxide structure and good oxide adherence. 850X

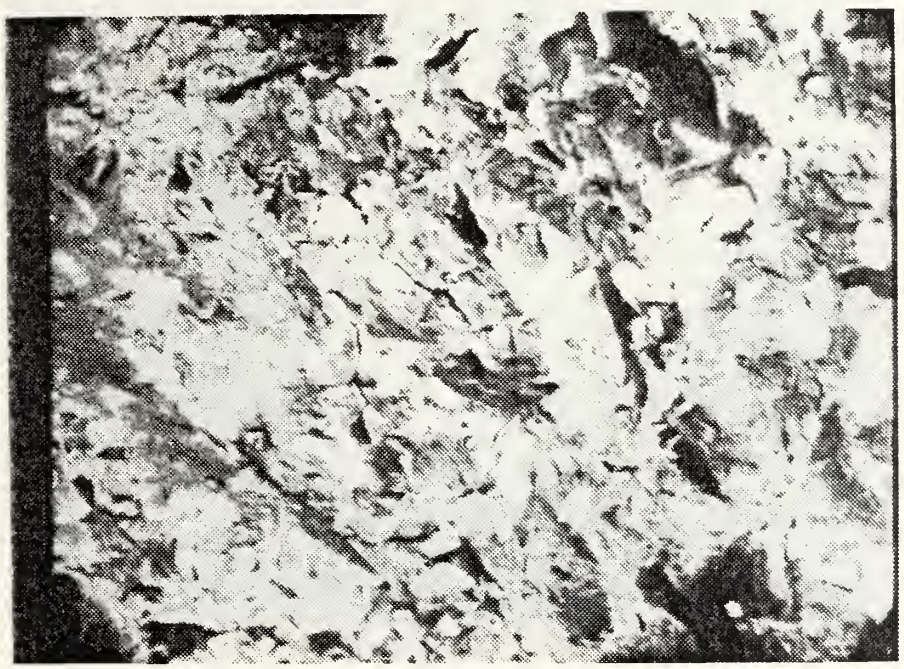

Figure B.25 Cracked and rough surface of a polished and heavily peened sample. SEM, 1000X

Figure B.26 Example of a surface from which most of the oxide has spalled. SEM, 45 deg. tilt, 100X

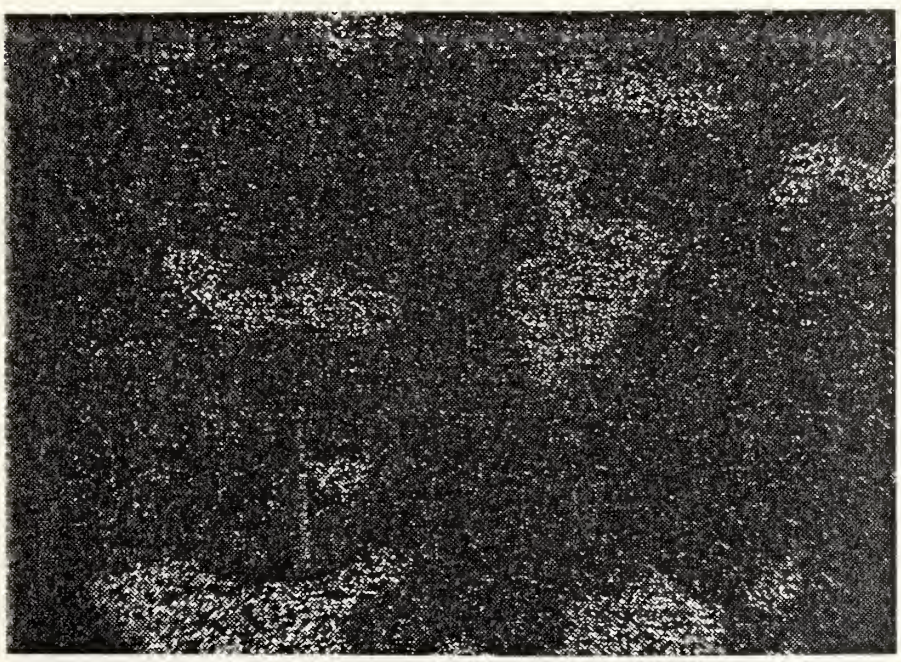

Figure B.27 EDAX/SEM scan for aluminum in the area shown in Figure 26. 100X

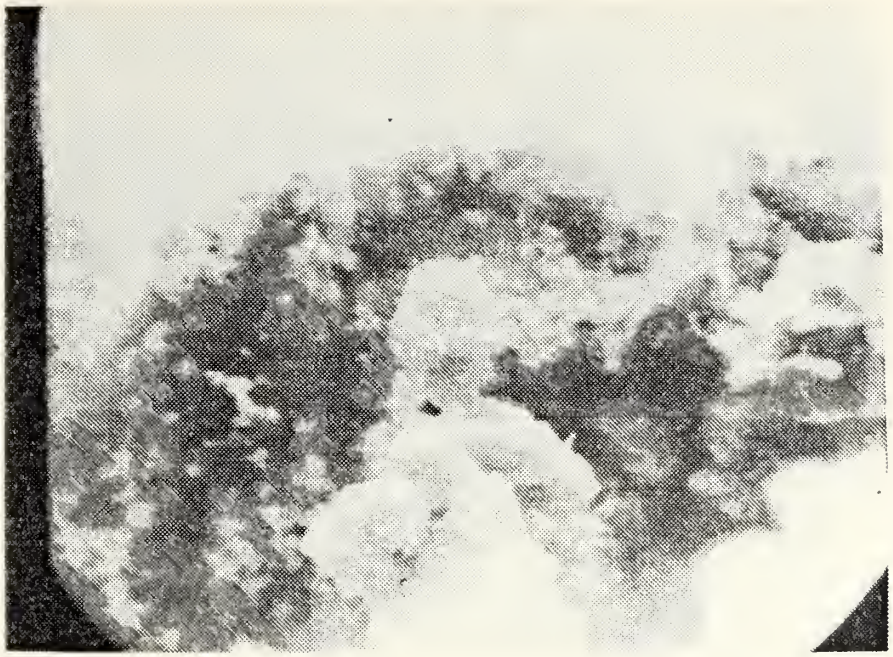

Figure B.28 Deep etched CoCrAl with Hf which shows the formation of micro-pegs which bond the oxide to the coating. SEM, 85 deg. tilt, 10000X

Figure B.29 Deep etched CoCrAl with Hf which shows the formation of micro-pegs which bond the oxide to the coating. SEM, 85 deg. tilt, 5000X

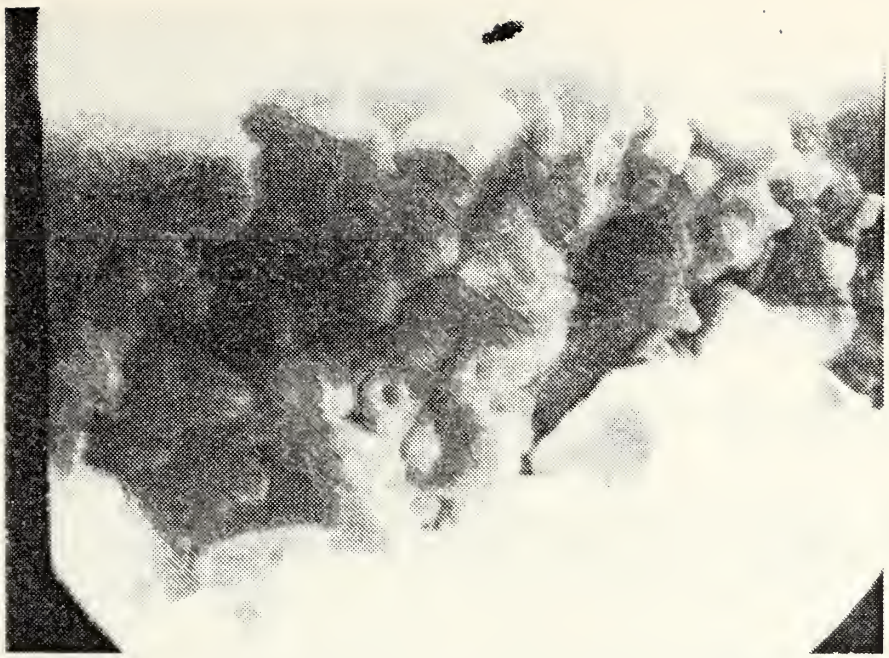

Figure B.30 Deep etched NiCrAl with La which shows no micro-peg formation. SEM, 85 deg. tilt, 5000X

Figure B.31 Deep etched CoCrAl with La which shows no micro-peg formation. SEM, 85 deg. tilt, 5000X

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